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# **Ferrographic and Spectrometer Oil Analysis From a Failed Gas Turbine Engine**

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# FERROGRAPHIC AND SPECTROMETER OIL ANALYSIS FROM A FAILED GAS TURBINE ENGINE

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## SUMMARY

An experimental gas turbine engine was destroyed as a result of the combustion of its Ti components. It was concluded that a severe surge may have caused interference between rotating and stationary compressor parts that either directly or indirectly ignited the Ti components. Several engine oil samples (before and after the failure) were analyzed with a Ferrograph, a plasma, an atomic absorption and an emission spectrometer to see if this information would aid in the engine failure diagnosis. The analyses indicated that a lubrication system failure was not a causative factor in the engine failure. Neither an abnormal wear mechanism nor a high level of wear debris was detected in the engine oil sample taken just prior to the test in which the failure occurred. However, low concentrations (0.2 to 0.5 ppm) of Ti were evident in this sample and samples taken earlier. After the failure, higher Ti concentrations ( $>2$  ppm) were detected in oil samples taken from different engine locations. Ferrographic analysis indicated that most of the Ti was contained in spherical metallic debris after the failure. Attempts to pinpoint the failure initiation site were inconclusive, but the oil analyses did eliminate a lubrication system bearing or shaft seal failure as the cause of the engine failure.

## INTRODUCTION

In 1976, an experimental gas turbine engine was being tested as part of the Full Scale Engine Research Program at the Lewis Research Center. While exploring a region of fan flutter, the engine appeared to encounter a stall, caught on fire and was destroyed.

It was concluded that the severe surge may have caused interference between rotating and stationary compressor parts that either ignited Ti by friction or caused structural failure with the resulting debris igniting the Ti.

The goals of engine oil analysis are to diagnose the condition of the oil wetted components (such as bearings and shaft seals) of an engine and to give early warnings of any future problems. A variety of different techniques (ref. 1) are being employed to effect these goals. These include spectrometric oil analysis (SOAP) using emission, atomic absorption, and plasma spectrometers. These devices measure the concentration of various elements in the oil (both dissolved and in particulate form) but are not able to distinguish among the various wear modes that can occur.

Another instrument, the Ferrograph, has been developed which magnetically separates wear debris from used lubricants onto a glass slide to yield a Ferrogram (refs. 2 to 4). Particles range from approximately 0.02 to a few micrometers and are arranged according to size on the slide. Individual particles

may be observed with a unique bichromatic microscope (the Ferroscope) or with a conventional scanning electron microscope.

The objective of this investigation was to determine if analyses of the engine lubricant using the Ferrograph and various spectrometers would provide any insight into the diagnosis of the above-mentioned engine failure.

Mr. Vernon Westcott of Foxboro Analytical, Burlington, Massachusetts, obtained the electron micrographs from the National Engineering Laboratory of Scotland, East Kilbride, Glasgow.

## APPARATUS AND PROCEDURE

### Ferrograph

The Ferrograph (refs. 2 to 4) is an instrument used to magnetically precipitate wear particles from a used oil onto a specially prepared glass slide. A mixture of 3 ml of used oil and 1 ml of solvent is prepared. This mixture is then slowly pumped over the slide. A solvent wash and fixing cycle follows which removes residual oil and permanently attaches the particles to the slide. The resulting slide with its associated particles is called a Ferrogram.

### Energy Dispersive X-ray Analysis

The elemental composition of the different types of wear debris was determined using energy dispersive X-ray analysis (EDX). In order to prevent charging in the scanning electron microscope, the Ferrogram slides were coated with either a thin layer ( $2 \times 10^{-8}$  m, 200 Å) of C or Au.

### Spectrometers

Basically, there are two methods for spectrometric oil analysis. One method uses the emission spectrograph where metallic atoms are excited by an electric arc to emit characteristic spectra. The intensity of these spectral lines is used to measure the metal concentration in the sample. The second method used the atomic absorption spectrophotometer where the sample is burned in a flame. The ground state atoms in the flame absorb a portion of a light beam transmitted through the flame. The amount of light absorbed is a measure of the metal concentration.

A recent innovation in emission spectrometers is the use of a "plasma" to excite the sample rather than the traditional arc or spark (ref. 5). This system provides a clear background, improved stability, and minimal matrix interference. The particle size independent technique (ref. 6) involved mixing the used lubricant with an acid-solvent mixture in order to dissolve the wear particles.

## RESULTS AND DISCUSSION

There were five oil samples (two prior to failure and three after failure) analyzed by various techniques. These included two engine oil samples dated May 27, 1976, and September 10, 1976 (before failure). Three other samples (after failure) were an engine oil sample dated September 14, 1976, a sample from the oil cooler and one from the oil filter.

Elemental analysis. - The above samples were spectroscopically analyzed for Fe, Ag, Al, Cr, Cu, Mg, Ni, Si, and Ti by routine emission spectrographic techniques (SOAP) (ref. 1) and by a plasma spectroscopic technique (ref. 5). Results for these analyses are shown in table I. In the two samples taken before the failure, low concentrations of Fe, Al, Cu, Si, and Ti were detected. There is nothing unusual in these results. In the September 14, 1976, engine oil sample taken after failure, there are definite increases in the concentrations of Fe, Al, Cr, Ni, Si, and Ti. Similar increases were noted for the two other "after failure" samples.

Since the ordinary emission spectroscopic techniques are somewhat dependent on the particle size of the debris present in the sample, a special atomic absorption technique (particle size independent) was used for Ti analysis (ref. 6). Analytical results for four samples using this technique appear in table II. Higher Ti concentrations were detected in all four samples compared to the plasma spectrometer results of table I. This technique is considered to be more accurate than the plasma emission analysis.

Ferrographic analysis (before failure). - Oil samples were also analyzed using the Ferrograph and selected areas of Ferrograms were further analyzed in a scanning electron microscope. An electron micrograph of wear debris from the engine oil sample (September 10, 1976) appears in figure 1(a). A typical particle (A) is analyzed by X-ray dispersive energy analysis (EDX) in figure 1(b). Particle A is predominantly Ti and Fe with lesser amounts of Cr, Mn, and Ni. It should be noted that in any small particle analysis using EDX, there may be surrounding particle interference. This means that contributions to some of the EDX peaks may be from surrounding or underlying particles or substrate since the analyzing depth or volume may, in some cases, be greater than the size of the analyzed particle.

The particle morphology of figure 1(a) is essentially that of normal rubbing wear particles (a benign form of wear) (refs. 7 and 8). However, there were spherical particles evident in the prefailure sample and an electron micrograph appears in figure 2(a). EDX analysis of the spheres appears in figure 2(b) and the background analysis of the glass Ferrogram appears in figure 2(c). The presence of Au is due to the evaporated Au film to prevent charging in the SEM. These spheres are essentially pure Fe. No other elements (besides the background elements) are detected by EDX. These large 10- to 15- $\mu$ m-diameter spheres are probably contaminants in the oil. Spherical particles have been related to bearing fatigue (refs. 7-10), but spheres from fatigue are usually much smaller (1 to 5  $\mu$ m in diameter) and have analyses similar to the bearing steel. Large spheres have been associated with cavitation erosion (ref. 8) and grinding (ref. 11).

Essentially the same area on the Ferrogram that appears in figure 2(a) is mapped for the distribution of Fe, Ti, Cu, and Si in figures 3(b) to (e). As can be seen, the majority of the particles contain Fe, while one particle contains Ti. Most of the Si and Cu is from the background.

Ferrographic analysis (after failure). - Figure 4 shows the type of particles present in the oil taken from the oil filter after the failure. As can be seen, the majority are spherical or spheroidal in nature. In reflected white light, the spherical particles have a variety of colors from white, to straw-colored, to deep blue. There is also evidence of tear-shaped or globular par-

ticles. These shapes, combined with the variety of colors, are indicative of a high-temperature melting and resolidification process. These particles are shown at higher magnification in figure 5.

Electron micrographs of debris from the engine oil sample (September 14, 1976) show similar particle types. These are shown in figure 6(a) with an accompanying EDX analysis in figure 6(b). The particles are predominantly Ti and Fe with lesser amounts of Cr and Ni.

Another micrograph of debris from engine sample (September 14, 1976) is shown in figure 7(a). X-ray distribution maps for Ti, Fe, Ni, Cr and Si appear in figures 7(b) through (f), respectively. In support of the previous figure, the debris consists of Ti and Fe with small amounts of Ni and Cr. The Si is essentially from the Ferrogram slide.

Wear severity index. - A parameter, the wear severity index, has been advocated by Bowen and Westcott (ref. 7). This parameter is calculated from optical density measurements made at the entry position of the Ferrogram ( $A_L$ ) and at the 50-mm position ( $A_S$ ). These are the areas covered by the large and small particles. Their sum ( $A_L + A_S$ ) yields the general level of wear, while their difference ( $A_L - A_S$ ) gives an indication of abnormality. These two quantities multiplied together  $(A_L + A_S)(A_L - A_S)$  yield the wear severity index  $A_L^2 - A_S^2$ , usually abbreviated  $I_S$ .

$I_S$  values for all of the oil samples, including the mean and standard deviation of six engine samples from 1974, are shown in figure 8. The  $I_S$  increased from about 10 to 140 from the May 27, 1976, sample to the September 10, 1976 sample. Although this is a large increase prior to failure, it is not significant when compared to the range of  $I_S$  values obtained from this engine in the past (i.e., 1974 samples). After failure,  $I_S$  values increase dramatically. This, of course, is expected. An especially large value was obtained from the oil filter sample.

Failure initiation site. - One of the more plausible failure modes was the ignition of Ti by friction between moving and stationary components in the high pressure compressor. There were two possibilities: (1) rotor/shroud source in which the Ti rotor blades would interfere with their shrouds (Metco 301, an abradable Ni-Cr-Fe alloy); and (2) a stator/spacer source in which the Ti knife edges on the spacers interfere with the labyrinth seal material (AMS 430, a stainless steel).

These types of failures would generate debris in the gas path of the engine. Conceivably, this debris could enter the bearing compartments through the seals and eventually find its way into the lubrication system.

It was hoped that analysis of this debris might indicate the location of the high speed rub. The nominal composition of the shroud material (Metco 301) is 69 percent Ni, 14 percent Cr, 8 percent Fe, 5.5 percent BN and 3.5 percent Al. The labyrinth seal material is AMS 430 which has a composition of 83 percent Fe, 17 percent Cr and 1 percent Mn. Examining many particles by EDX after failure (such as in fig. 7) was inconclusive. No analysis exactly matched the above compositions. This is not surprising considering the manner in which the

particles were formed. In addition, the particle analysis from figure 6(b) is very similar to the analysis of the particle in figure 1(b) which was from a sample taken prior to the failure. There definitely were Ti-containing particles in the engine lubricant prior to the engine failure.

#### CONCLUDING REMARKS

It was concluded that a failure in the lubrication system was not involved and, therefore, did not contribute to the engine failure. Attempts to pinpoint the possible failure initiation site in the high pressure compressor were inconclusive.

#### SUMMARY OF RESULTS

The Ferrograph, an emission spectrometer, a plasma spectrometer and an atomic absorption spectrophotometer were used to analyze engine oil samples from a failed gas turbine engine. The major results are summarized as follows:

1. No abnormal wear mechanisms nor high levels of wear were detected in engine oil samples taken prior to engine failure.
2. However, low concentrations of Ti (0.2 to 0.5 ppm) were evident in the engine oil prior to failure. Higher Ti concentrations (>2 ppm) were detected after failure.
3. Ferrographic analysis indicated that most of the Ti was contained in spherical metallic debris after the failure. Some Ti debris was present before the failure.

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TABLE I. - ELEMENTAL ANALYSIS OF OIL SAMPLES USING ORDINARY  
SOAP PROCEDURE AND A PLASMA SPECTROMETER

| Sample                         |  | Elements, ppm |       |       |       |       |       |       |        |       |
|--------------------------------|--|---------------|-------|-------|-------|-------|-------|-------|--------|-------|
|                                |  | Fe            | Ag    | Al    | Cr    | Cu    | Mg    | Ni    | Si     | Ti    |
| 5/27/76<br>Before<br>failure   | Soap <sup>a</sup><br>(plasma<br>spect.) <sup>b</sup> | 1             | 0     | 0     | 0     | 2     | 0     | c     | 5      | 0     |
|                                |  | (0.5)         | (0.1) | (0.5) | (0.1) | (1.1) | (0.2) | (0.1) | (2.2)  | (0.2) |
| 9/10/76<br>Before<br>failure   | Soap<br>(plasma)                                     | 1             | 0     | 0     | 0     | 2     | 0     | c     | 4      | 0     |
|                                |  | (0.5)         | (0)   | (0.3) | (0.1) | (0.6) | (0.1) | (0)   | (0.8)  | (0.3) |
| 9/14/76<br>After<br>failure    | Soap<br>(plasma)                                     | 5             | 0     | 4     | 2     | 3     | 0     | c     | 0      | 2     |
|                                |  | (2.5)         | (0)   | (1.6) | (0.9) | (0.6) | (0)   | (0.9) | (10.6) | (2.2) |
| Oil Cooler<br>after<br>failure | Soap<br>(plasma)                                     | 3             | 0     | 3     | 0     | 3     | 0     | c     | 5      | 2     |
|                                |  | -             | -     | -     | -     | -     | -     | -     | -      | -     |
| Oil Filter<br>after<br>failure | Soap<br>(plasma)                                     | -             | -     | -     | -     | -     | -     | -     | -      | -     |
|                                |  | (4.1)         | (0)   | (2.6) | (1.0) | (1.6) | (0.5) | (1.2) | (49.3) | (3.1) |

<sup>a</sup>Army Oil Analysis Lab, Ft. Campbell, KY

<sup>b</sup>Air Force Materials Lab, WPAFB, OH 45433

<sup>c</sup>Not determined

TABLE II. - ANALYSIS OF OIL SAMPLES FOR TITANIUM  
USING PARTICLE SIZE INDEPENDENT ATOMIC  
ABSORPTION SPECTROPHOTOMETRY<sup>c</sup>

| Sample       | Titanium concentration,<br>ppm       |
|--------------|--------------------------------------|
| From engine: |                                      |
| 5/27/76      | 0.4 <sup>a</sup> (0.2)               |
| 9/10/76      | 0.5 <sup>a</sup> (0.3)               |
| 9/14/76      | 3.1 <sup>a</sup> (2.2)               |
| Oil Filter   | <sup>b</sup> 11.5 <sup>a</sup> (3.1) |

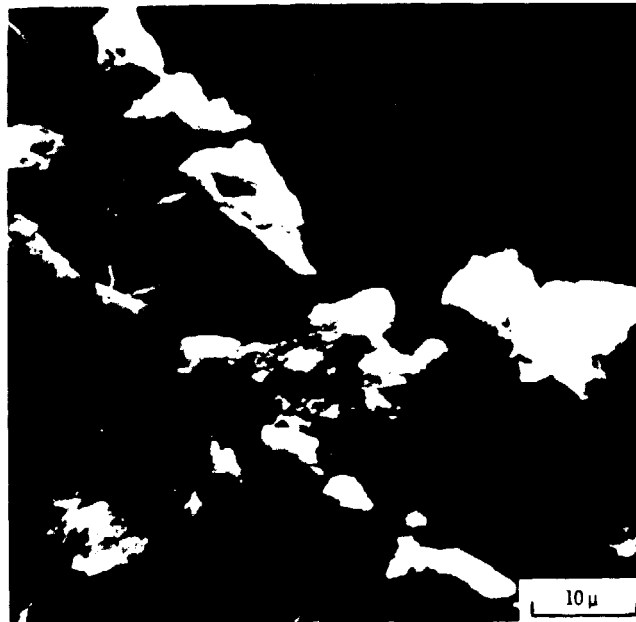
<sup>a</sup>plasma data from table 1.

<sup>b</sup>Sample contained large particles which settled  
to bottom of bottle.

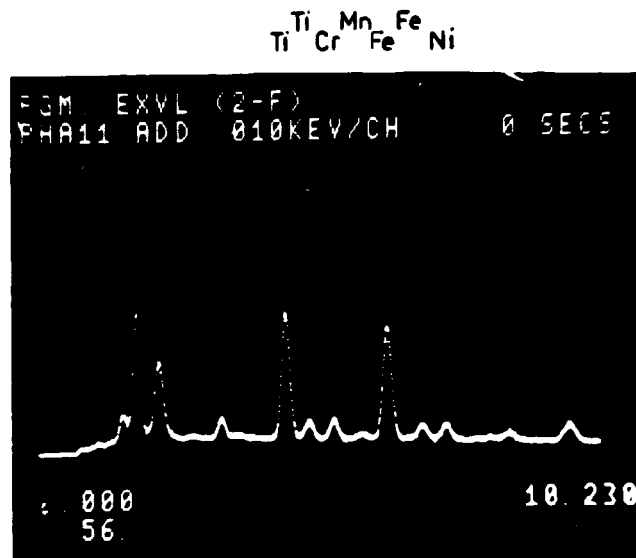
<sup>c</sup>Reference 6.



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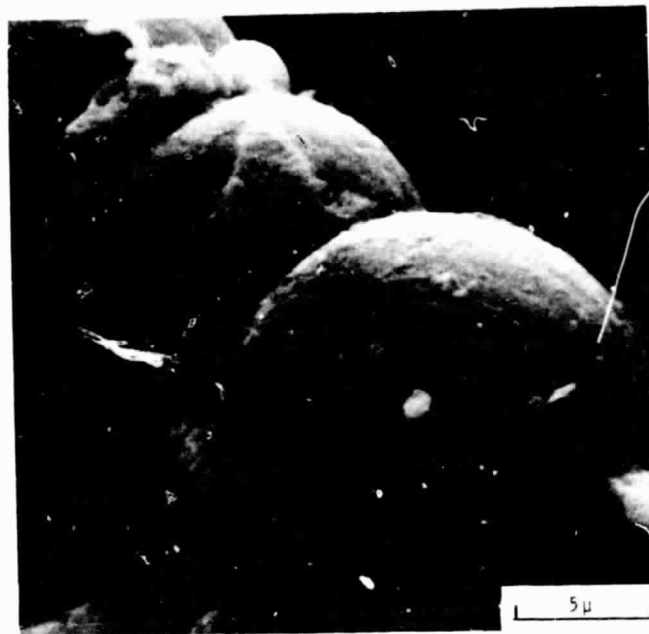
(a) Electron micrograph of wear debris.



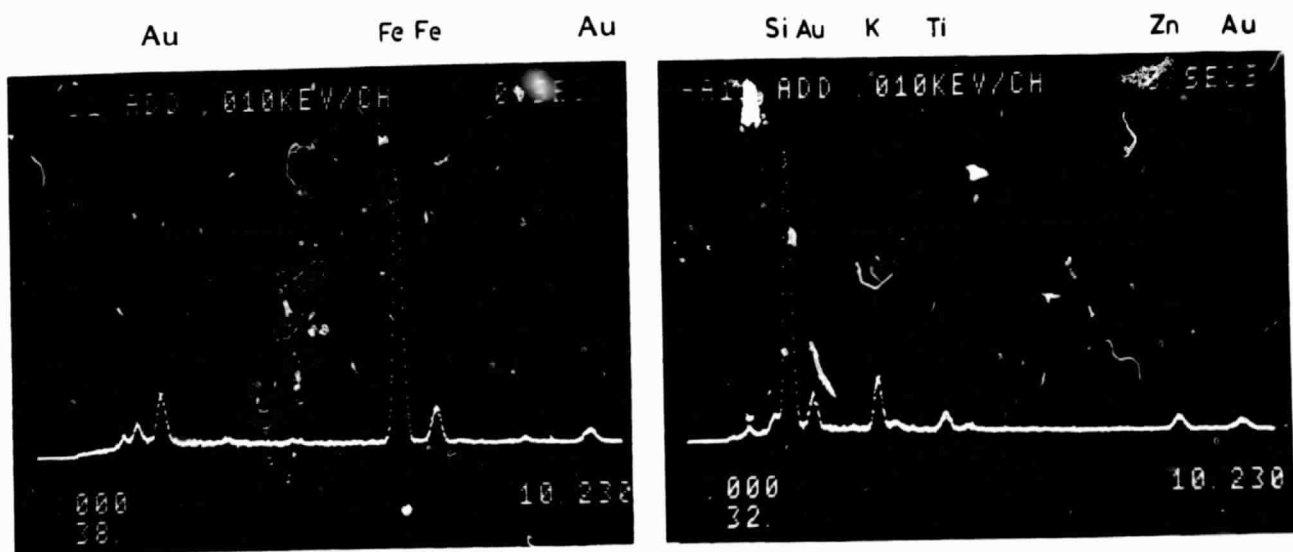
(b) EDX analysis of particle A.

Figure 1. - Wear debris from oil sample 9/10/76 (before failure).

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(a) Electron micrograph of spherical particles.

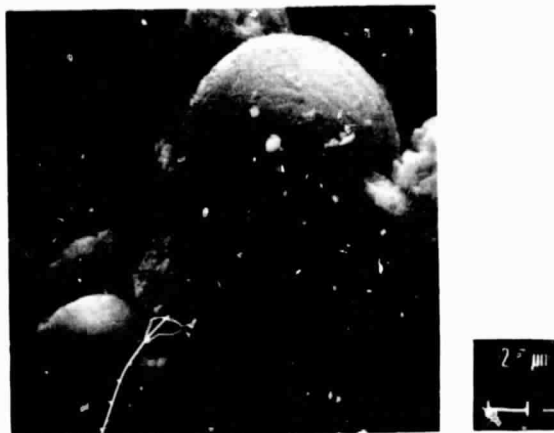


(b) Energy dispersive X-ray analysis (EDX) of spheres.

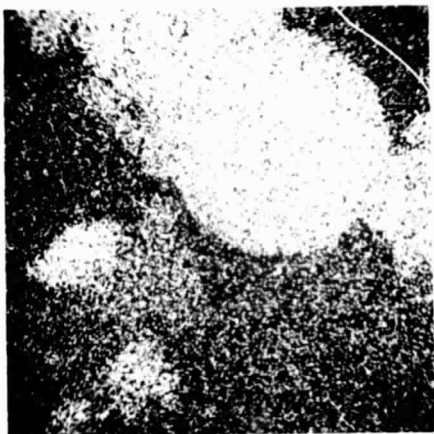
(c) Background EDX of ferrogram.

Figure 2. - Spherical particles from oil sample 9/10/76 (before failure).

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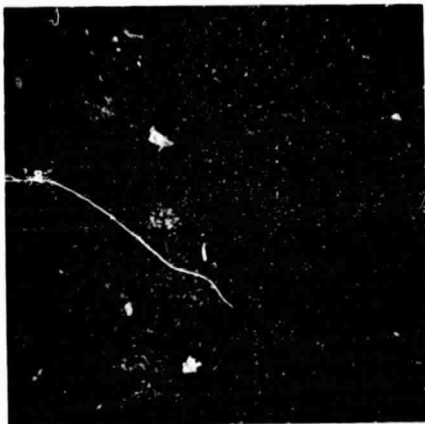
(a) Electron micrograph of spherical particles and debris



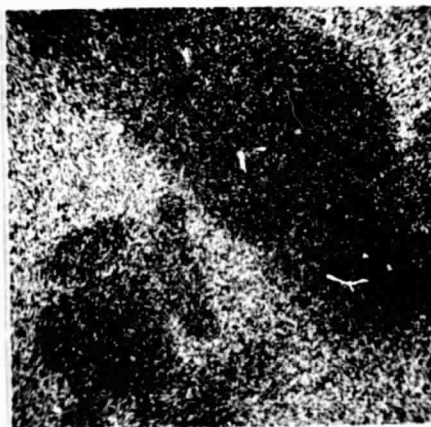
(b) X-ray distribution of Fe.



(c) X-ray distribution of Ti.



(d) X-ray distribution of Cu.



(e) X-ray distribution of Si.

Figure 3. - Spherical particles and debris from oil sample 9/10/76 (before failure) and accompanying elemental X-ray maps.

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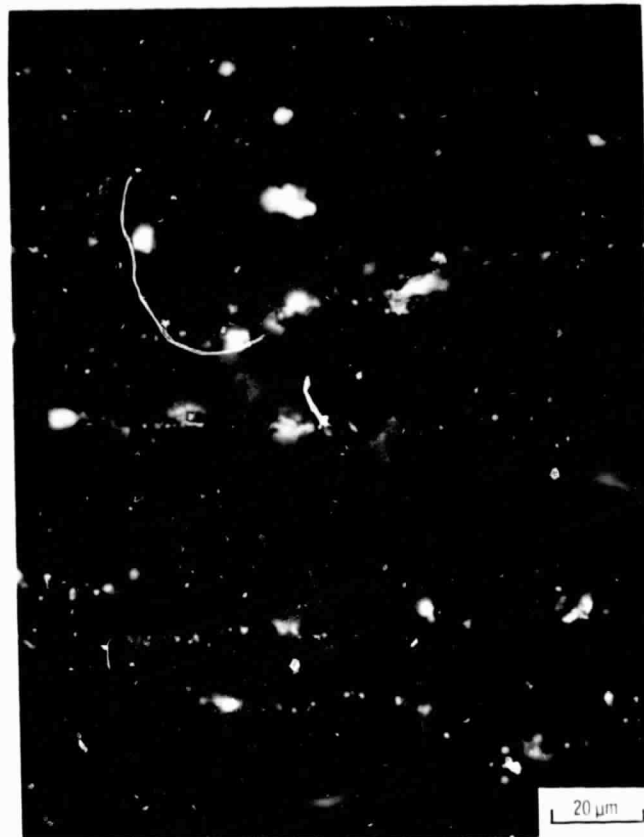


Figure 4. - Optical micrograph of spherical particles and debris from oil in oil filter (after failure).

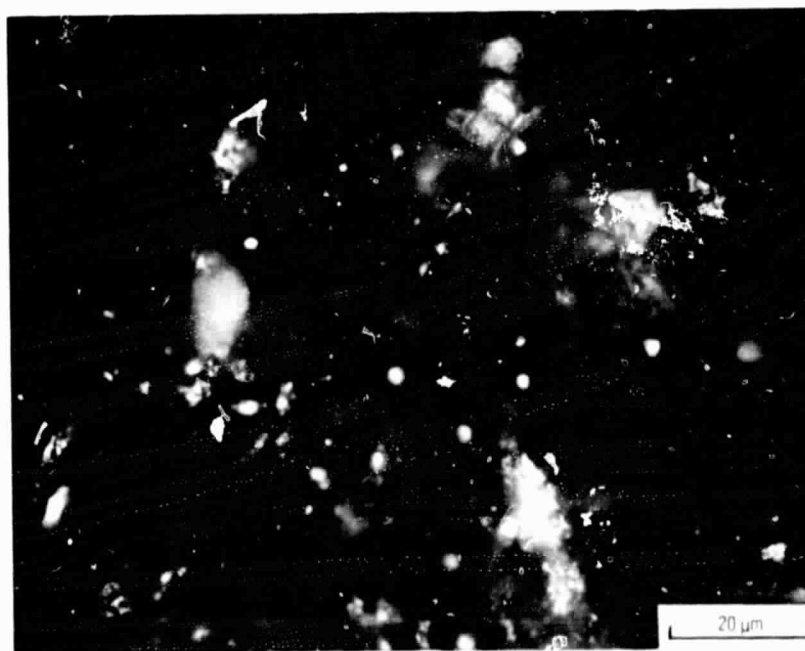
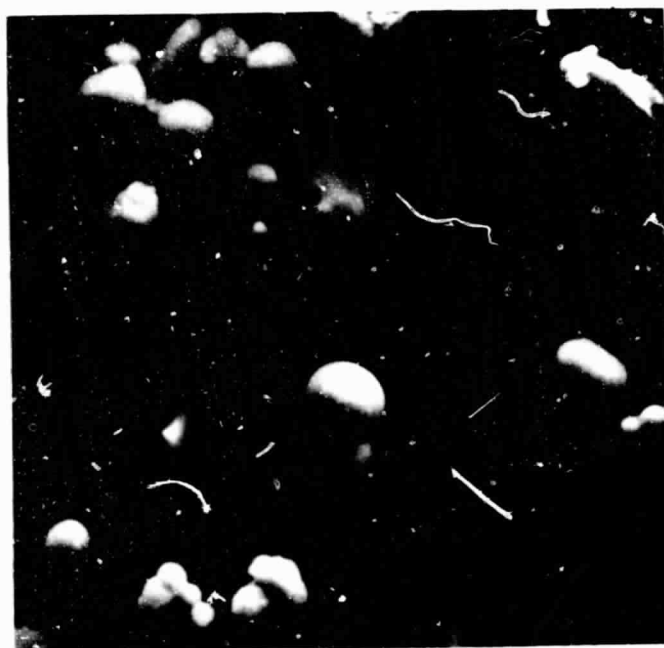
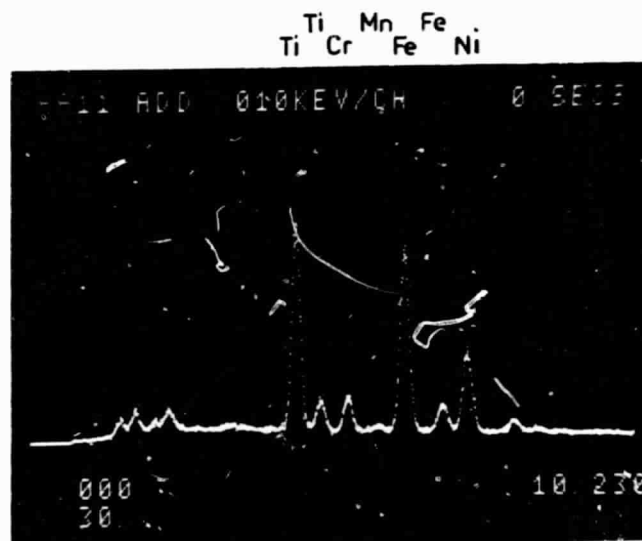


Figure 5. - Debris from oil sample from oil filter (after failure).



(a) Electron micrograph of debris.

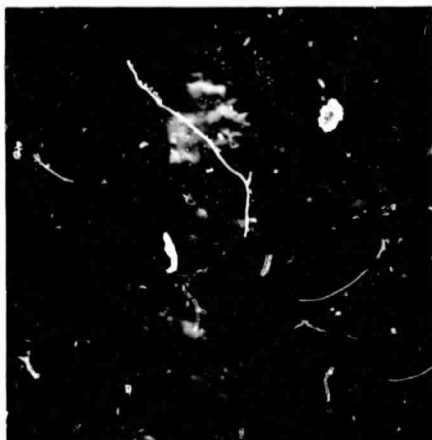


(b) Typical EDX analysis of particles.

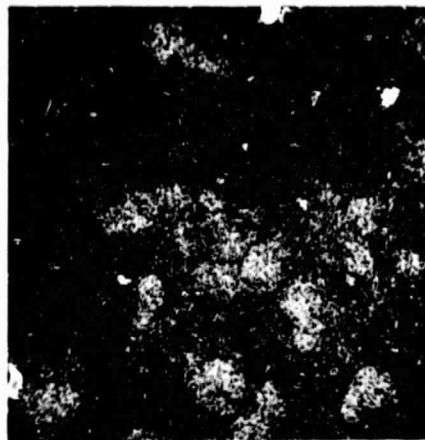
Figure 6. - Debris from oil sample 9/14/78 (after failure).

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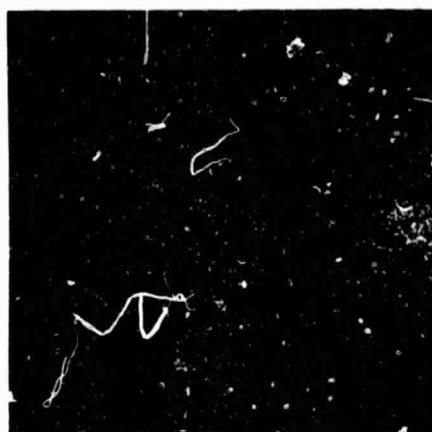
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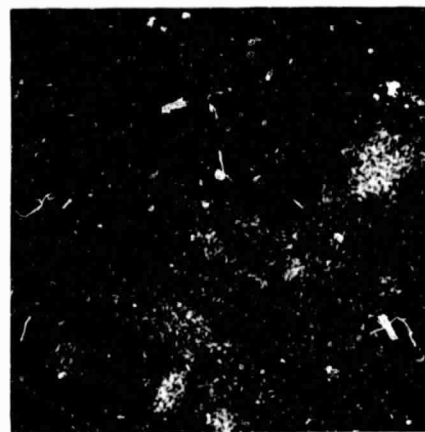
(a) Electron micrograph of debris.



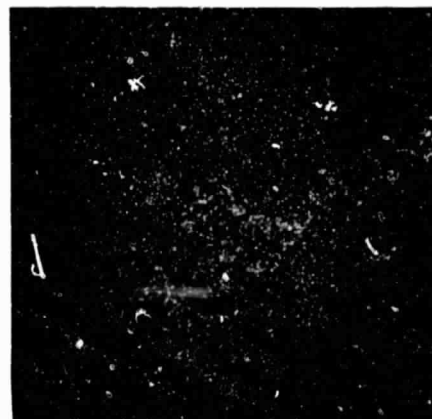
(b) X-ray distribution of Ti.



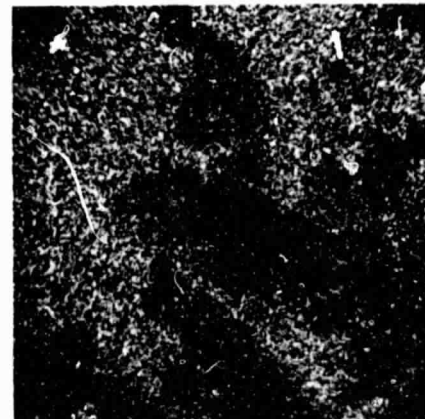
(c) X-ray distribution of Fe.



(d) X-ray distribution of Ni.



(e) X-ray distribution of Cr.



(f) X-ray distribution of Si.

Figure 7. - Debris from oil sample 9/14/76 (after failure) and accompanying elemental X-ray maps.

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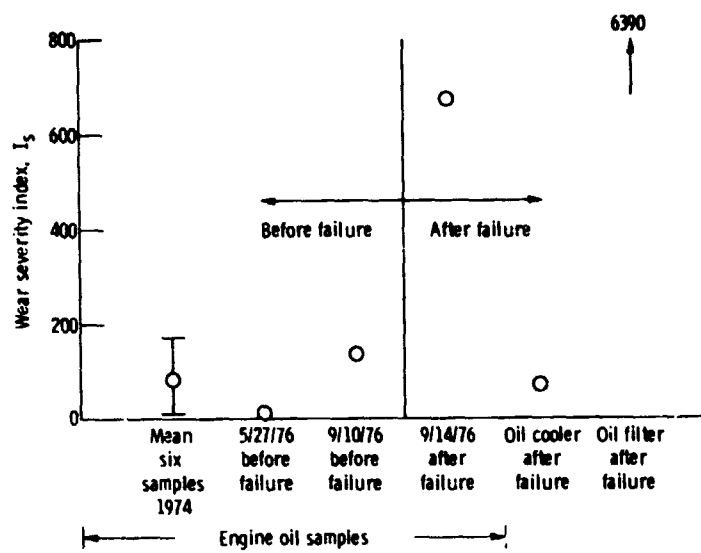


Figure 8. - Wear severity index ( $I_s$ ) for various oil samples.